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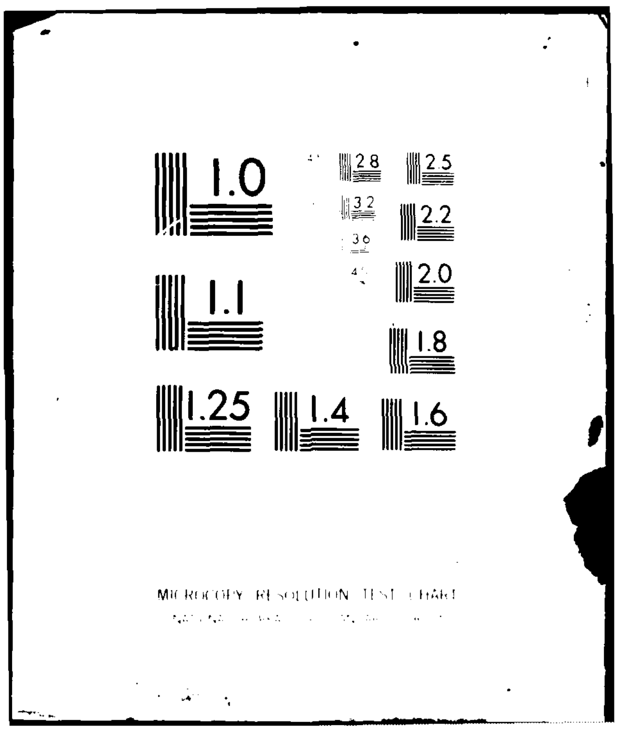
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DEPARTMENT OF DEFENCE

DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION

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MELBOURNE, VICTORIA

TECHNICAL NOTE

MRL-TN-436

RECRYSTALLISED RDX FOR RDX/POLYETHYLENE WAX COMPOSITIONS

William S. Wilson

Approved for Public Release

MAY, 1980

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ABSTRACT

The impact and shock sensitivities of RDX/polyethylene wax compositions prepared from RDX Grade A Class 1 (recrystallised from cyclohexanone) have been measured using the Rotter Impact Test and Gap Test respectively, and the results have been compared with those obtained for compositions prepared from RDX Grade B Class 1 (milled and boiled). The increased impact sensitivity and decreased shock sensitivity of the compositions based on the recrystallised RDX are attributed to the presence of occluded cyclohexanone and the increased particle size.

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16. ABSTRACT (if this is security classified, the announcement of this report will be similarly classified):

The impact and shock sensitivities of RDX/polyethylene wax compositions prepared from RDX Grade A Class 1 (recrystallised from cyclohexanone) have been measured using the Rotter Impact Test and Gap Test respectively, and the results have been compared with those obtained for compositions prepared from RDX Grade B Class 1 (milled and boiled). The increased impact sensitivity and decreased shock sensitivity of the compositions based on the recrystallised RDX are attributed to the presence of occluded cyclohexanone and the increased particle size.

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RECRYSTALLISED RDX FOR RDX/POLYETHYLENE WAX COMPOSITIONS

1. INTRODUCTION

In a recent report (MRL-R-722) the formulation and sensitivity of a series of RDX/polyethylene wax compositions were described, together with the compaction of these compositions and the shock sensitivity and velocity of detonation of the resultant charges [1]. A programme of work was then initiated to examine the effects of inert diluents and their physical properties, such as density, hardness and particle size, on the properties of this class of explosive [2]. These explosive compositions were based on a polyethylene wax designated AC 629 and manufactured by Allied Chemicals Ltd., as the binder-*cum*-sensitiser, and RDX Grade B Class 1 [3] as the explosive component.* The penultimate step in the manufacture of this grade of RDX was a milling and boiling process, included to break down crystalline aggregates and to remove the last traces of acid remaining from the preparative stage.

Production of RDX Grade B Class 1 ceased in 1977, and all RDX manufactured in Australia since that time has been recrystallised from cyclohexanone to give RDX Grade A Class 1** with a particle size distinctly larger than that of the material previously available. It has long been recognised that the particle size of an explosive can drastically alter its sensitivity to such stimuli as impact and shock waves. Therefore, to provide a data base from which to examine the effect of particle size of an inert additive (carborundum) on the properties of this class of explosives,

* In MRL-R-722 this material was incorrectly termed RDX Grade A Class 1. This error arose through confusion with the previous description of the explosive as RDX Grade 1A.

** Throughout this report RDX Grade A Class 1 and RDX Grade B Class 1 will be referred to as recrystallised RDX and milled and boiled RDX respectively.

a series of RDX/polyethylene wax compositions was prepared using recrystallised RDX, tested for impact sensitivity and then pressed into pellets to examine the sensitivity to shock initiation of these explosives and its dependence on composition and density. The results obtained previously for compositions based on milled and boiled RDX are included for comparison, and an attempt has been made to account for differences in terms of RDX particle size.

Most of the RDX produced in Australia is used in Composition B for filling bombs and projectiles, and an investigation is currently in progress to assess the effects of using recrystallised RDX in this explosive [4]. It was hoped that results presented here would also be relevant to that investigation.

2. RDX PARTICLE SIZE DISTRIBUTION

RDX Grade B Class 1 (milled and boiled) was, and RDX Grade A Class 1 (recrystallised from cyclohexanone) is, produced at Albion Explosives Factory by the Woolwich process (direct nitration of hexamine) and both conform to the Australian Defence Standard DEF (AUST) 5382. The characteristics which show differences between the two grades of explosive are listed in Table 1 below.

TABLE 1

EXTRACT FROM RDX SPECIFICATION DEF (AUST) 5382

No.	Characteristic	Grade A Class 1	Grade B Class 1
4	Acidity		
	(a) Total acidity as HNO_3, %	0.015 max	0.05 max
	(b) Occluded acidity as HNO_3, %	0.01 max	0.035 max
8	Cyclohexanone, %	0.2 max	-
11	Particle Size Distribution		
	Retained on 850 μm AS Sieve, %	Nil	Nil
	Retained on 500 μm AS Sieve, %	2.0 max	2.0 max
	Retained on 300 μm AS Sieve, %	25 max	25 max
	Total passing 75 μm AS Sieve, %	12 max	60 max

For reasons of safety RDX is normally stored in Australia as a slurry in water (ca 40% w/w). At MRL this water is removed prior to use by filtration followed by drying to constant weight at 70°C. The dry RDX is packaged in 1 kg lots. Duplicate 25 g samples of dried recrystallised RDX were taken from five such packages using the coning and quartering technique. Each sample was slurried in water containing a little detergent and subjected to wet sieve analysis. The slurry was poured into the coarsest sieve, which was immersed in water until the solid was just covered and the wet RDX was passed through the sieve by gentle agitation by hand for ten minutes. The material retained on the sieve was transferred to a weighed Gooch crucible and dried to constant weight at 65°C. The RDX passing through the sieve was transferred to the next sieve, and the process was repeated. Sieving losses were found to be in the range 1-2%. Results of the ten sieve analyses are shown in Table 2, together with an average particle size distribution for recrystallised RDX. These data are also shown in histogram form in Figure 1, together with data obtained by Eadie and Milne for the milled and boiled RDX [5].

Data from sieve analyses may be treated statistically in a number of ways to generate values descriptive of the particle *size distribution.*

A *Weight Average* is given by

$$\bar{\mu}_w = \sum w_i \bar{\mu}_i \Big/ \sum w_i$$

where w_i is the weight of a sieve cut and $\bar{\mu}_i$ is the average particle diameter of that cut, while a *number average* is given by

$$\bar{\mu}_n = \sum n_i \bar{\mu}_i \Big/ \sum n_i$$

where

$$n_i = w_i \Big/ \frac{4}{3} \pi \rho \left(\frac{\bar{\mu}_i}{2} \right)^3$$

In this treatment more importance is placed on the finer particles. Alternatively the *median* size of the sample, the mid-point in the distribution where half the particles are larger and half the particles are smaller can be interpolated from a plot of cumulative weight per cent undersize (or oversize). These values were determined for the recrystallised RDX, and are presented in Table 3 together with the corresponding values obtained for milled and boiled RDX [5].

It is interesting to note, however, that the two grades of RDX both have bulk powder densities of about 1.18 Mg/m^3 (experimental values of 1.184 and 1.177 Mg/m^3 were obtained for the recrystallised and milled and boiled materials respectively).

TABLE 2

PARTICLE SIZE DISTRIBUTION OF RDX GRADE A CLASS 1

RDX Sample	< 75 µm	75-106 µm	106-212 µm	212-300 µm	300-425 µm	425-500 µm
1A	2.40	4.88	33.24	49.01	7.18	3.29
1B	0.59	4.07	32.26	54.62	7.61	0.85
2A	0.97	1.58	32.13	56.32	8.46	0.54
2B	0.90	2.31	28.57	60.08	4.28	3.89
3A	1.27	3.41	31.42	55.07	8.03	0.08
3B	1.62	3.44	30.55	56.74	7.48	0.17
4A	0.96	2.23	28.29	57.72	10.10	0.70
4B	1.36	3.11	30.49	54.68	9.97	0.39
5A	1.07	2.95	30.45	55.71	9.11	0.71
5B	1.49	4.05	33.34	54.11	6.66	0.36
AVERAGE	1.26	3.20	31.07	55.41	7.89	1.10

TABLE 3

VALUES FOR RDX PARTICLE SIZE DISTRIBUTIONS

	Grade A Class 1 (Recrystallised)	Grade B Class 1 (Milled and Boiled)
Weight Average	227 μm	95 μm
Number Average	89 μm	45 μm
Median	236 μm	75 μm

Photomicrographs of the two grades of RDX using oil as dispersant are shown in Figure 2. The recrystallised RDX is seen as discrete, well-formed particles, while the milled and boiled RDX contains a large number of much smaller crystals which tend to be present as large aggregates. RDX has the same refractive index as bromoform, and photomicrographs were also prepared using that liquid as dispersant in an attempt to examine internal discontinuities in the RDX. However as Figure 3 shows, these efforts were unrewarded, as no conclusive evidence was obtained.

3. PREPARATION OF RDX/POLYETHYLENE WAX COMPOSITIONS

RDX/polyethylene wax compositions with nominal wax content 1-15% by weight were prepared from recrystallised RDX and the emulsifiable polyethylene wax AC 629 manufactured by Allied Chemicals Ltd., following the AWRE wax emulsion process described previously [1]. Briefly, an emulsion of the wax in water, oleic acid and morpholine was added to a slurry of the RDX in water, and the emulsion was broken by the addition of dilute sulphuric acid to the hot mixture (95°C). After washing with distilled water to remove all traces of acid and with 0.05% aqueous methyl *p*-hydroxybenzoate to inhibit fungal attack of the polyethylene wax, the explosive was dried to constant weight at 70°C. The RDX/polyethylene wax compositions were submitted for analysis of wax content, and the results given in Table 4.

4. IMPACT SENSITIVITY

As a prelude to compaction into consolidated charges, the RDX/polyethylene wax compositions prepared from the recrystallised RDX were tested in the MRL Rotter Impact Machine using the 5 kg weight. For each composition a sequence of 50 caps was tested following the Bruceton 'Staircase' procedure to determine the 50% 'explosion' height, which was compared with the 50% 'explosion' height for a standard grade of RDX to

TABLE 4

ANALYSIS AND IMPACT SENSITIVITY OF RDX/POLYETHYLENE WAX COMPOSITIONS PREPARED FROM RECRYSTALLISED RDX (GRADE A CLASS 1)

Nominal Composition	RDX (%)	Wax (%)	Figure of Insensitiveness*	Mean Gas Vol. (ml)
Pure RDX	100.0	0.0	77	18
99:1	99.0	1.0	90	19
98:2	98.0	2.0	115	17
97:3	96.9	3.1	121	12
96:4	95.8	4.2	133	12
95:5	94.4	5.6	128	10
94:6	94.2	5.8	128	9
93:7	92.7	7.3	131	5
92:8	91.5	8.5	129	5
91:9	91.6	8.4	135	3
90:10	89.6	10.4	135	3
89:11	88.2	11.8	137	2
88:12	87.3	12.7	140	2
87:13	86.8	13.2	140	2
86:14	85.3	14.7	140	2
85:15	84.2	15.8	149	1

* Impact sensitivity is normally given to the nearest 10 F of I units. To enable the variation with composition to be observed more clearly, the values are quoted here to the nearest F of I unit.

which a Figure of Insensitiveness (F of I) of 80 was assigned. The criterion defining an 'explosion' was the evolution of not less than 1 ml of gases recorded by a special gas measuring burette.

The F of I for RDX/polyethylene wax compositions prepared from recrystallised RDX, and the mean volumes of gas evolved on impact testing are also given in Table 4, and are illustrated graphically in Figures 4 and 5 respectively together with the corresponding data obtained previously for compositions prepared from milled and boiled RDX [1]. It is noted that, although the two grades of RDX themselves have the same impact sensitivity (F of I 80), the explosives formulated from recrystallised RDX have a lower F of I - i.e., they are more sensitive to impact - than are those prepared from milled and boiled material, at least for compositions containing more than about 5% of wax. For the two compositions 85:15 the difference is a substantial 50 F of I units.

It is difficult to account for such sensitivity differences in terms of the principal difference between the two forms of RDX, namely the particle size distribution. Indeed it might be expected that the recrystallised RDX, having a larger particle size and therefore a smaller specific surface area, should have fewer potential hot spot sites at which initiation of explosive reaction could occur, and that the inclusion of wax should desensitise it more efficiently than the finer milled and boiled RDX. This is clearly not the case. An alternative explanation involves the physical location of the crystal imperfections (potential hot-spot sites) which contribute to the sensitivity of the explosive. In the milled and boiled RDX, in which the grains are broken and more irregular, the potential hot-spot sites are on the exterior of the crystals and are progressively coated and desensitised by the addition of wax. On the other hand the recrystallised RDX crystals are more regular and contain fewer of these external defects, but they may contain significant numbers of solvent inclusions (see Table 1) which cannot be desensitised by *any* quantity of wax. However, as has already been discussed, attempts to demonstrate the presence of such solvent inclusions in photomicrographs of RDX dispersed in bromoform were unsuccessful.

By way of comparison it may be noted from the data in Figure 5 that the mean volume of gas evolved on impact testing of the RDX/polyethylene wax compositions was the same for the two grades of RDX. Thus, if the hypothesis of Mortlock and Wilby is accepted [6], although the ease of initiation of reaction in RDX/polyethylene wax depends on the RDX from which it was made, the ease of propagation of that reaction appears not to do so.

5. COMPACTION OF RDX/POLYETHYLENE WAX COMPOSITIONS

The RDX/polyethylene wax compositions were pressed into 2.5 g pellets 12.7 mm (0.50 in) in diameter and about the same length, in order to measure the variation in shock sensitivity of these explosives as a function of both density and composition (wax content). The pellets were pressed individually in a nest of five moulds, using as a press an 'Instron' Universal Testing Machine Model TT-CM operating in the compression mode with an FRM-Type load cell. For all experiments the lowest possible crosshead speed, viz., 0.5 mm min^{-1}, was used throughout the actual application of the pressing load, although higher speeds were used to move the crosshead into the required position. For all pressings a two minute dwell time (the period of time over which the pressing load is applied to the explosive) was adopted.

RDX/polyethylene wax prepared from recrystallised RDX to the nominal composition 92:8 (actually containing 8.5% wax) was pressed into pellets using the method described above, with the pressing load being varied between 3.92 kN and 12.75 kN. The densities of the pellets so produced are given in Table 5 below, and are also shown in graphical form in Figure 6 together with corresponding results obtained for compositions based on milled and boiled RDX [1]. Throughout the range of loads and densities under consideration the compositions formulated from recrystallised RDX were more difficult to press, requiring larger loads to achieve the same densities, and the resultant pellets being somewhat more friable.

TABLE 5

DENSITY OF RDX/POLYETHYLENE WAX 92:8 PELLETS

Pressing Load (kN)	Density (Mg/m^3)	Voidage (%)
3.92	1.530	8.55
4.90	1.553	7.27
5.88	1.572	6.04
6.86	1.582	5.44
8.83	1.598	4.48
10.79	1.610	3.77
12.75	1.615	3.47

It has already been noted that the two forms of RDX have essentially the same bulk density of about 1.18 Mg/m^3 and the difference in ease of pressing is therefore a reflection of the RDX particle size distributions. Presumably the compaction of compositions formulated from milled and boiled RDX is achieved predominantly by a redistribution of the wax and the RDX particles, whilst the larger particles of the recrystallised RDX tend to be crushed and broken up during the compaction process. This was born out when RDX/polyethylene wax 92:8 based on each form of RDX was soxhlet-extracted with carbon tetrachloride to remove the wax and the particle size distribution of the residual RDX was determined by sieve analysis. Each composition was analysed both unpressed and after pressing under a load of 5.8 kN. The particle size of the milled and boiled RDX was unaffected by incorporation of wax or compaction. However, the recrystallised RDX, although unchanged by the incorporation of wax, was reduced in particle size by the compaction process, as is reflected by the data in Table 6.

TABLE 6

PARTICLE SIZE OF RECRYSTALLISED RDX (GRADE A CLASS 1)

RDX Sample	Weight Average	Number Average	Median
As received	227 μm	89 μm	236 μm
Incorporated & extracted	229 μm	86 μm	236 μm
Incorporated, pressed & extracted	200 μm	50 μm	227 μm

RDX/polyethylene wax compositions formulated from recrystallised RDX containing between 2 and 15% by weight of wax were pressed into pellets using the method and conditions described above. The pressing loads were selected to produce explosive pellets of constant voidage 4.7%; however as the results in Table 7 indicate, the experimentally determined mean voidage was 5.2%.

TABLE 7

DENSITY OF RDX/POLYETHYLENE WAX PELLETS OF VARYING WAX CONTENT

Wax Content (%)	Pressing Load (kN)	Density (Mg/m^3)	Voidage (%)
2.0	16.67	1.677	5.24
4.2	12.75	1.652	4.91
5.8	8.24	1.616	5.66
8.5		1.584	5.20
10.4	4.90	1.551	5.71
12.7	4.61	1.530	4.96
14.7	4.02	1.500	5.04

6. SHOCK SENSITIVITY

The sensitivity of an explosive to initiation by shock waves is measured at MRL using an adaptation of the Gap Test described by Cachia and Whitbread [7] and illustrated in Figure 7. Briefly, a standard detonator (the Scale 1 Gap Test Donor, comprising an exploding bridgewire to initiate a low density PETN pellet) generates a standard shock which is attenuated by a stack of laminated 0.05 mm brass shims 25 mm square. The attenuated shock wave strikes the receptor or test explosive, usually a cylindrical pellet 12.7 mm (0.50 in) in diameter and 25.4 mm (1.00 in) long, which rests on a mild steel witness block. If on firing the assembly, a deep, sharply defined dent is produced in the witness block, the test explosive is said to have detonated. The critical gap, the thickness of brass required to prevent 50% of detonations in the test explosive, is determined by the Bruceton "staircase" procedure, in which the gap thickness is increased or decreased by an increment of 0.10 mm (0.05 mm if the critical gap is less than 1 mm) depending on whether the previous firing produced a detonation or a failure. Normally 25 'shots' are fired after the approximate median point has been established, although fewer can be used if the quantity of test explosives available so dictates.

This method was used to determine the shock sensitivity of various RDX/polyethylene wax charges prepared from recrystallised RDX. Two pellets 12.7 mm (0.50 in) long stacked end-on-end were used for each receptor charge. The results of these tests are found in Table 8.

Data from Table 8 are also presented graphically in Figures 8 and 9, which illustrate the variation with density and composition respectively of the shock sensitivity of RDX/polyethylene wax prepared from recrystallised RDX, together with the corresponding relationships for the compositions based on milled and boiled RDX [1]. As might be anticipated, the compositions prepared from the recrystallised RDX show the same trends, namely that the shock sensitivity of this class of explosive shows a steady increase with increasing density and a steady decrease with increasing wax content. However the compositions prepared from the recrystallised RDX in general are significantly less sensitive to shock than are those based on milled and boiled RDX. Thus, as Figure 8 indicates, at densities below about 1.56 Mg/m^3 (93% theoretical maximum density) RDX/polyethylene wax 92:8 prepared from recrystallised RDX is markedly less sensitive than the explosive prepared from the milled and boiled RDX, although the shock sensitivities of the two explosives do not appear to be significantly different above this density. Further, Figure 9 reveals that, when they are pressed to about 94.8% of theoretical maximum density, the compositions based on recrystallised RDX with varying proportions of wax are consistently significantly less sensitive than those compositions prepared from milled and boiled RDX. This reduction in shock sensitivity can be attributed to the increase in RDX particle size, and parallels similar results obtained for pressed TNT [8] and for various experimental HMX formulations [9]. The increase in particle size reduces the surface area of the explosive, and consequently reduces the number of potential hot spots present on that surface at which explosive can be initiated. It also reduces the surface area available and exposed for the transfer of energy from the reaction gases to unreacted explosive during propagation. (It should be noted,

TABLE 8

SHOCK SENSITIVITY OF RDX/POLYETHYLENE WAX COMPOSITIONS

PREPARED FROM RECRYSTALLISED RDX (GRADE A CLASS 1)

Wax Content (%)	Density (Mg/m^3)	Relative Density (% TMD)	Voidage (%)	Critical Barrier Thickness (mm)	Standard Deviation (mm)
8.1	1.539	91.94	8.06	0.957	0.022
8.1	1.553	92.77	7.23	1.253	0.022
8.1	1.572	93.91	6.09	1.563	0.015
8.1	1.582	94.50	5.50	1.676	0.019
8.1	1.598	95.46	4.54	1.659	0.042
8.1	1.610	96.18	3.82	1.711	0.017
8.1	1.615	96.48	3.52	1.744	0.050
2.0	1.677	94.76	5.24	2.807	0.062
4.2	1.652	95.09	4.91	2.405	0.021
5.8	1.616	94.34	5.66	2.014	0.036
(8.1	1.584	94.85	5.25	1.575	-*)
10.4	1.551	94.29	5.71	0.927	0.020
12.7	1.530	95.05	4.95	0.564	0.008
14.7	1.500	94.96	5.04	0.323	0.009

* Interpolated from Figure 8.

however, that an increase in particle size in *low* density explosive charges can lead to an increase in shock sensitivity. Thus low density PETN compacts, at 55% theoretical maximum density, show a small but steady increase in shock sensitivity with increasing particle size [10]).

7. CONCLUSIONS

A series of RDX/polyethylene wax compositions containing 1-15% by weight of wax has been prepared from RDX Grade A Class 1 (recrystallised from cyclohexanone), and the impact and shock sensitivities of these explosives have been examined using the Rotter Impact Test and the Gap Test respectively. The compositions show the same general trends as those prepared from RDX Grade B Class 1 (milled and boiled), in that the impact sensitivity of the powders decreases steadily with increasing wax content, while the shock sensitivity increases with increasing density and decreases with increasing wax content. However, there are differences in detail between the two series of compositions. The compositions prepared from the recrystallised RDX are more sensitive to impact than those based on milled and boiled RDX, at least for wax contents greater than 5%. They are also more difficult to press, in that greater loads are required to achieve the same densities, and the resultant pellets are more friable. Pressed RDX/polyethylene wax charges prepared from recrystallised RDX are generally less sensitive to shock initiation than are those based on milled and boiled RDX. These differences have been attributed to occluded solvent (cyclohexanone) present in the recrystallised RDX, and the greater particle size of that material.

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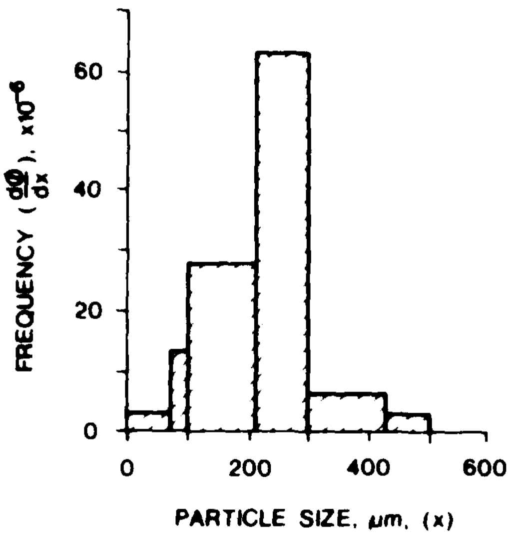
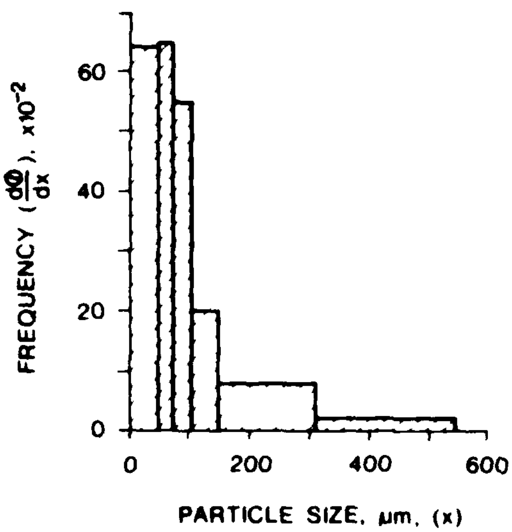


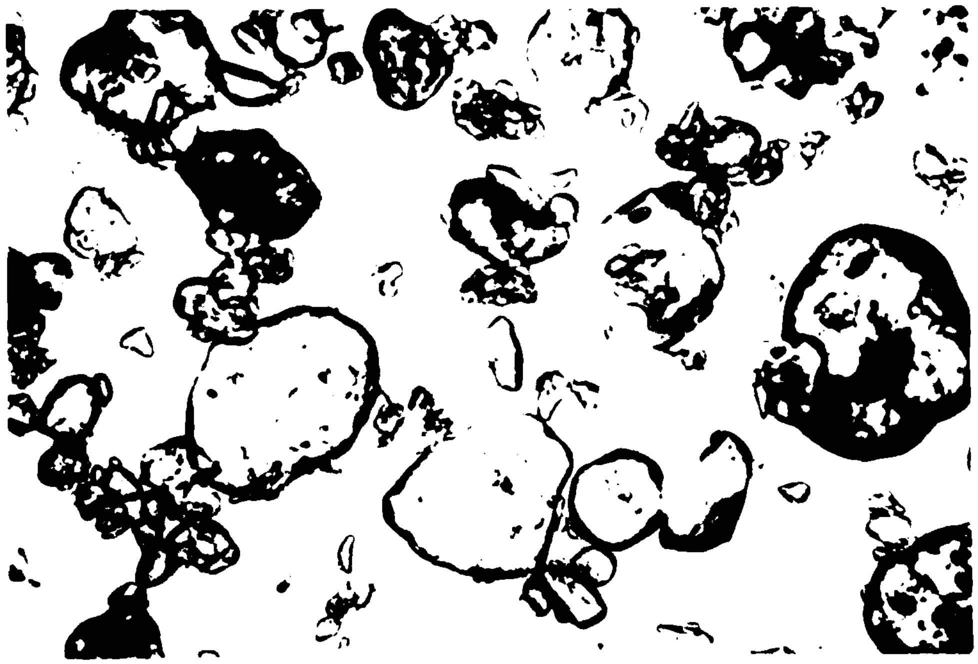
(i) RDX Grade A Class 1 (recrystallised from cyclohexanone).



(ii) RDX Grade B Class 1 (milled and boiled), from ref. 5.

FIG. 1 - RDX Particle Size Distributions

(a) RDX Grade A Class 1

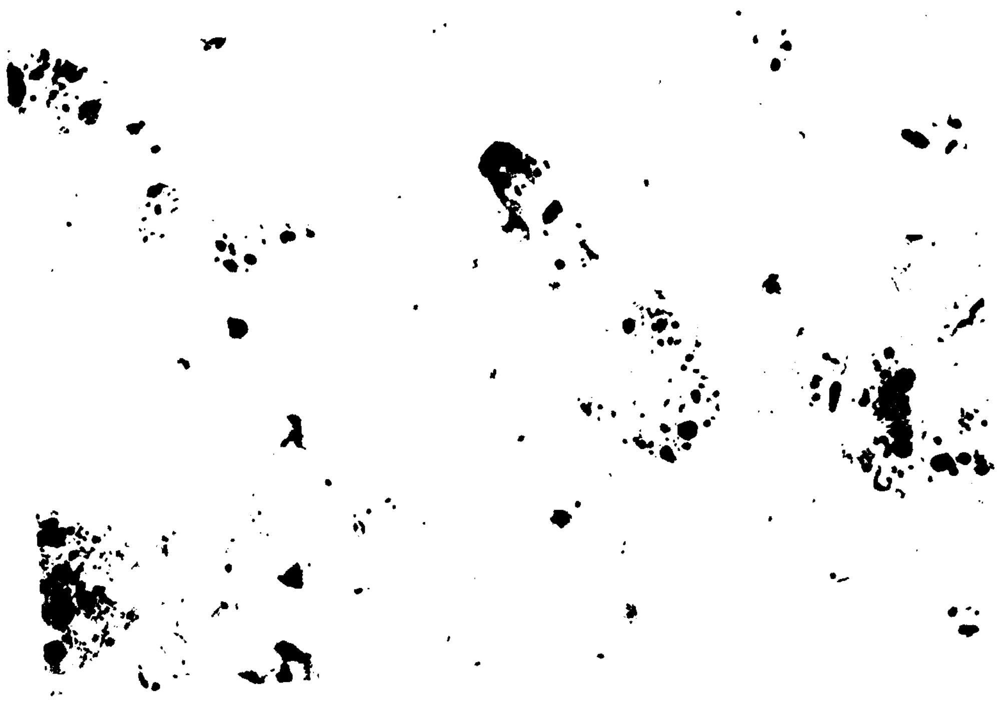


(b) RDX Grade B Class 1

1 mm

FIG. 2 - Photomicrographs of RDX Grades

(a) RDX Grade A Class 1



(b) RDX Grade B Class 1

1 mm

FIG. 3 - Photomicrographs of RDX Grades Dispersed in Bromoform

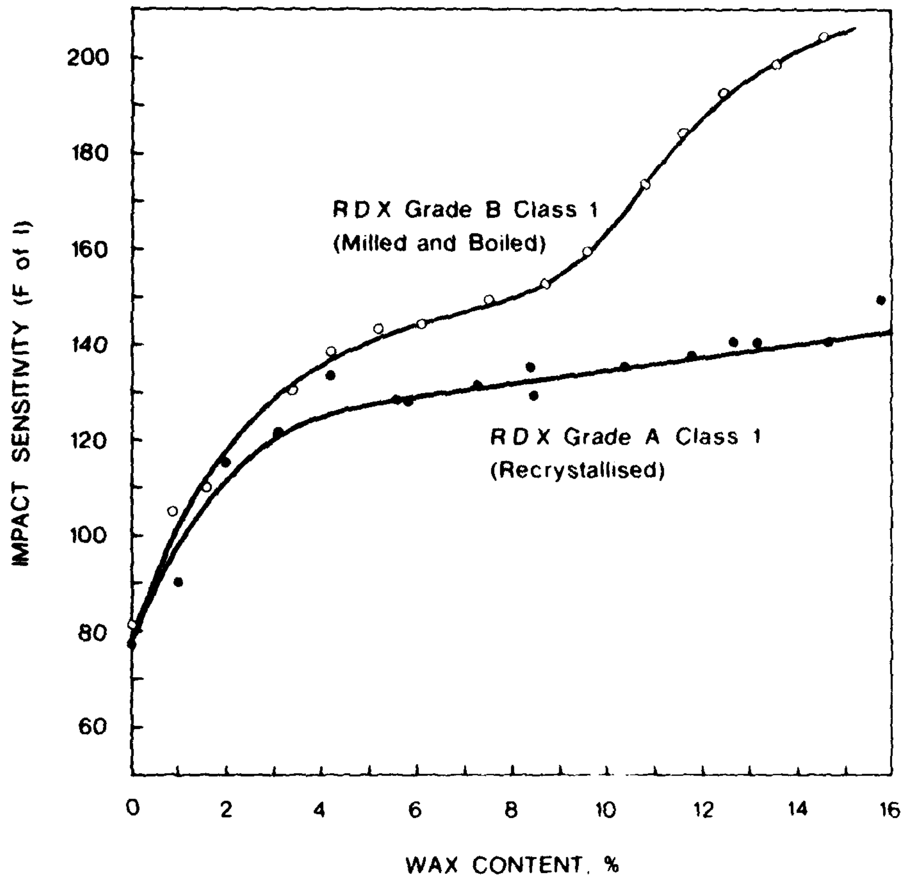


FIG. 4 - Impact Sensitivity of RDX/Polyethylene Wax Compositions as a function of RDX Type and Wax Content.

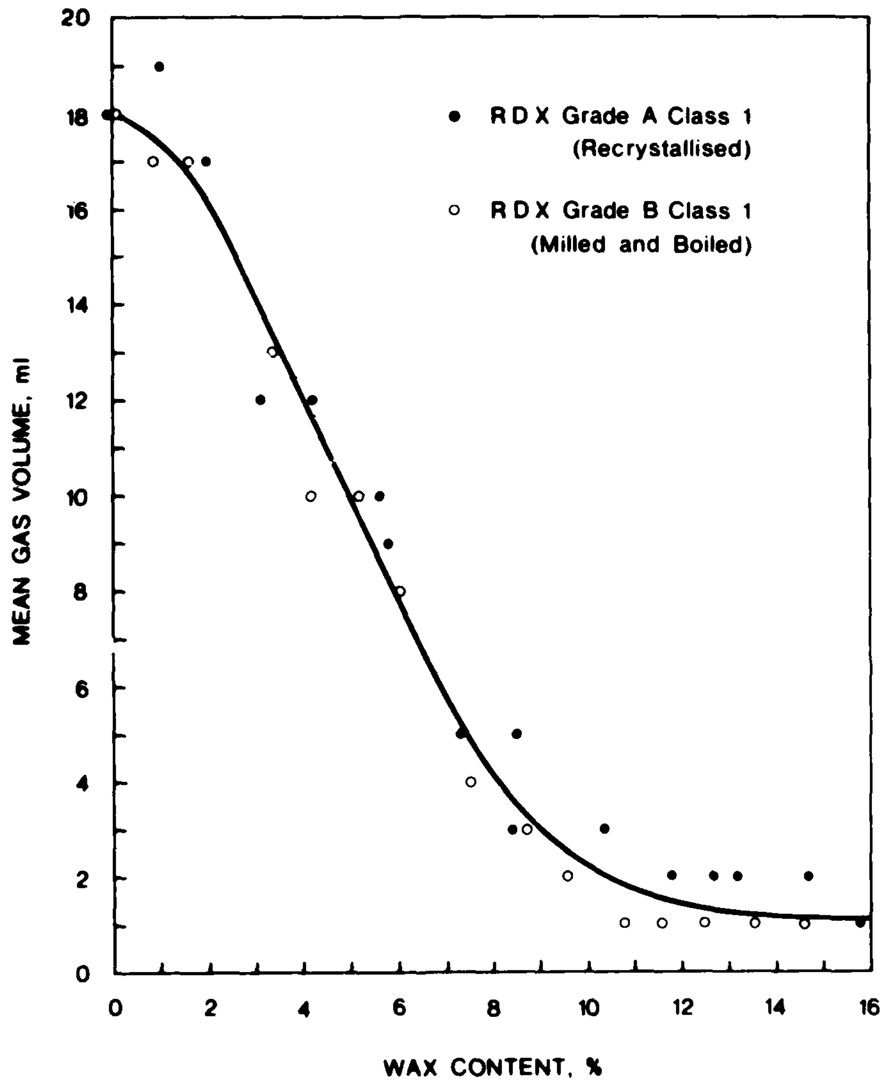


FIG. 5 - Mean Volume of Gas evolved on Impact Testing of RDX/Polyethylene Wax Compositions as a function of RDX Type and Wax Content.

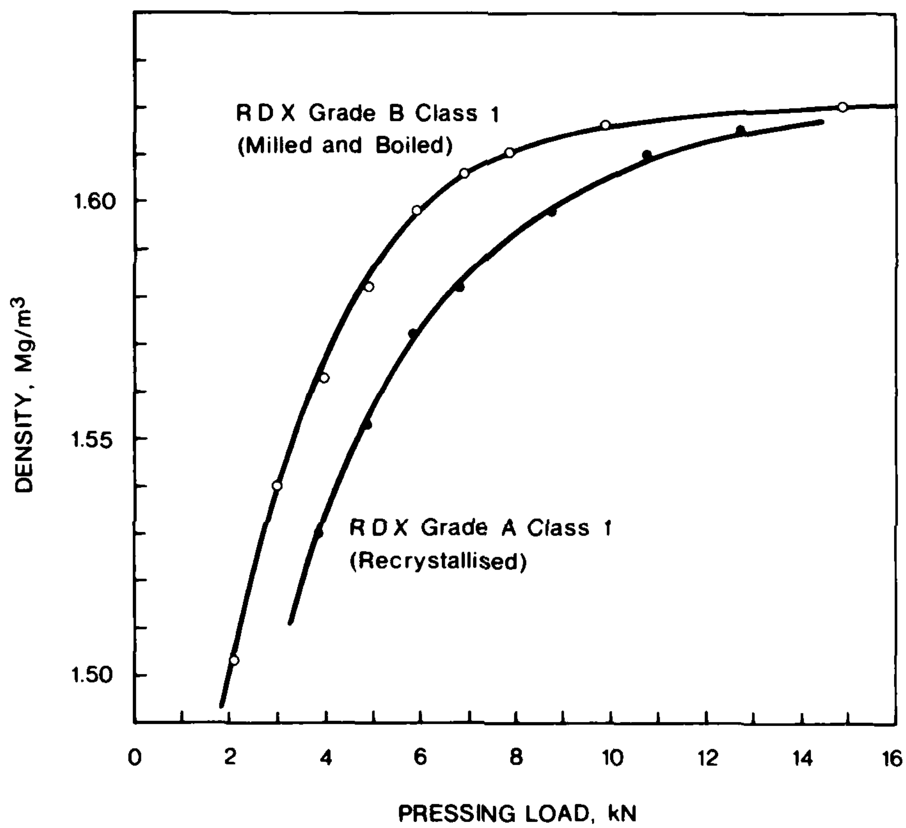


FIG. 6 - Density of RDX/Polyethylene Wax 92:8 as a function of Pressing Load.

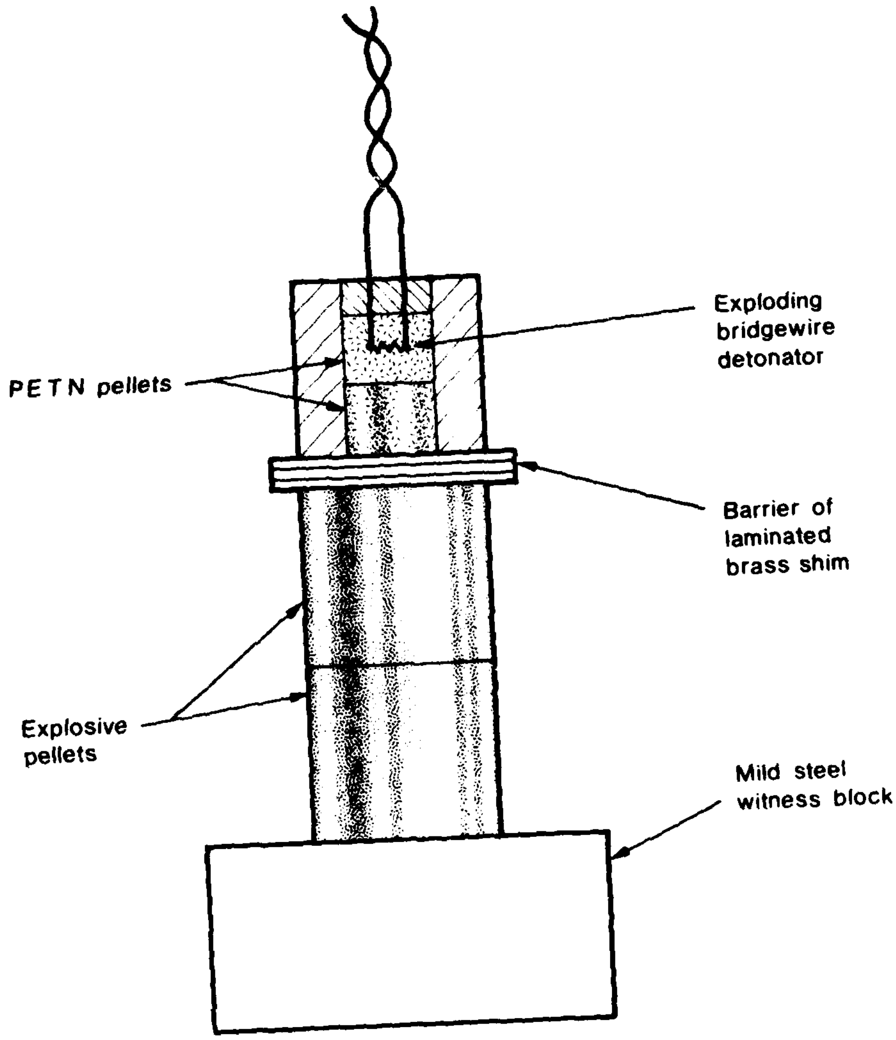


FIG. 7 - Gap Test Assembly for Measurement
of Shock Sensitivity.

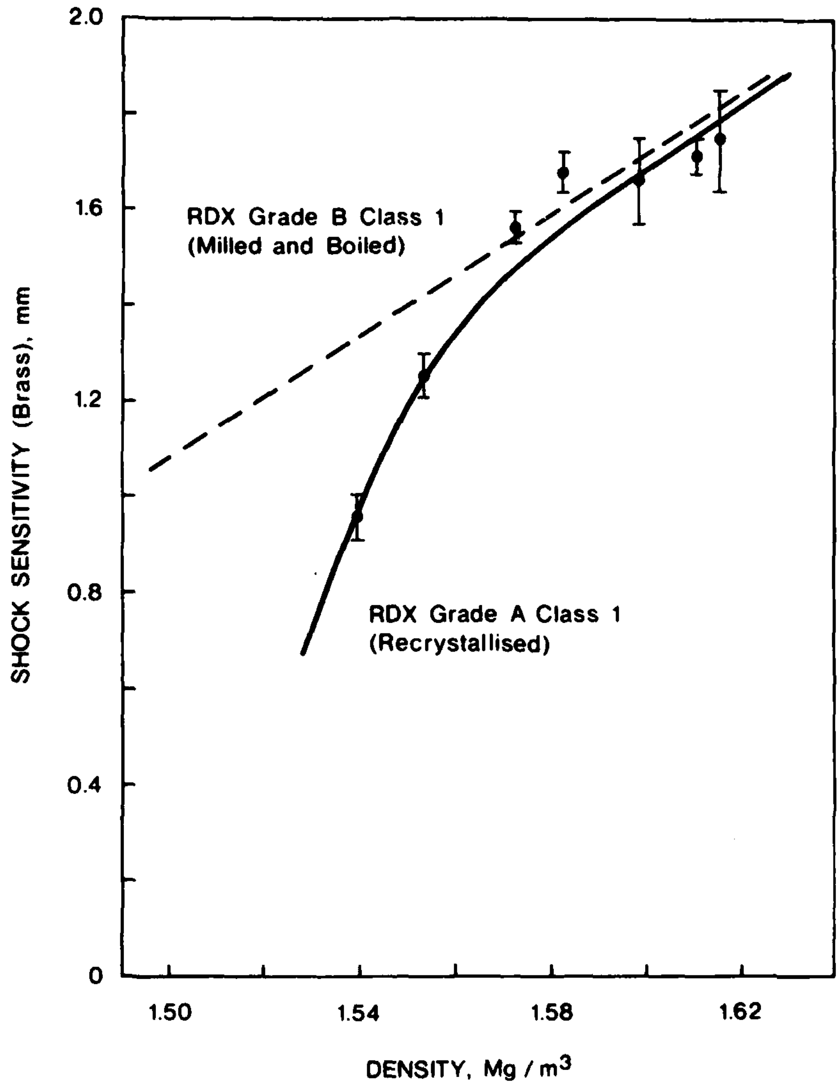


FIG. 8 - Shock Sensitivity of RDX/Polyethylene Wax 92:8 as a function of Density.

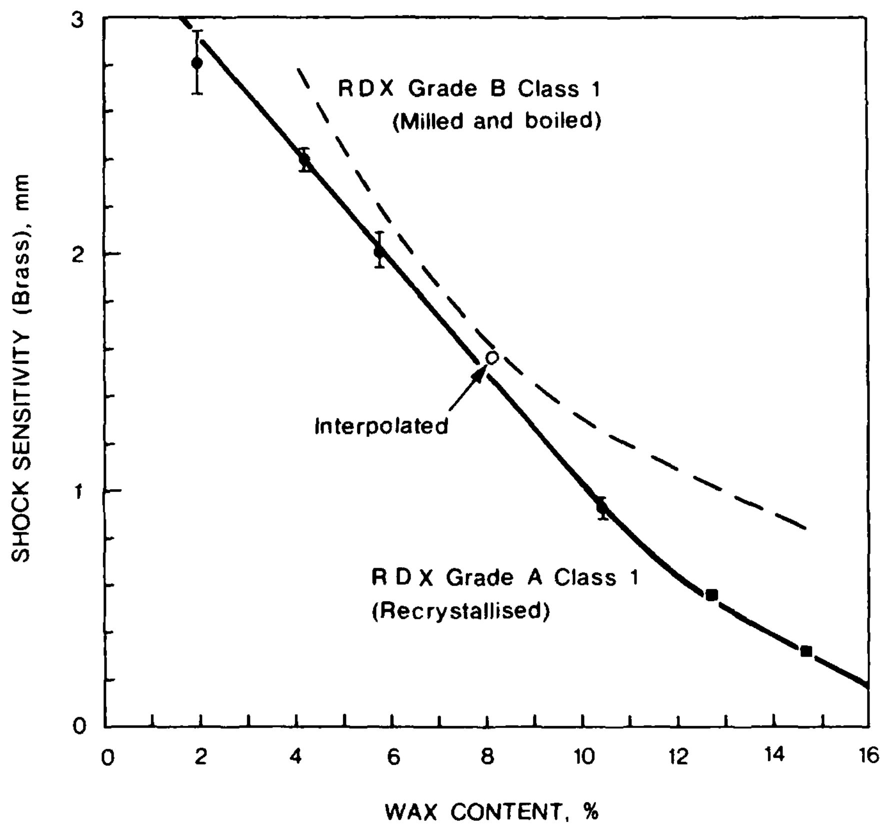


FIG. 9 - Shock Sensitivity of RDX/Polyethylene Wax, pressed to 95% Theoretical Maximum Density, as a function of Composition.

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